# Adapting a triple-axis spectrometer for small angle neutron scattering measurements

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**Abstract**. Small angle neutron scattering (SANS) instruments typically require a cold source and a large spread of wavelengths in order to enhance incident flux. However, as a direct result of the unavailability of a cold source at the Canadian Neutron Beam Centre (CNBC), we have resorted to adapting a triple-axis spectrometer for SANS measurements. This is achieved through the use of multiple converging incident beams which enhance the neutron flux on the sample by a factor of 20, compared to a single beam of same spot size. Furthermore, smearing effects due to vertical divergence from the slit geometry are reduced through the use of horizontal Soller collimators. As a result, this modified triple-axis spectrometer enables SANS measurements to a minimum $q$ value ($q_{\min}$) of 0.006 $Å^{-1}$ [1]. Data obtained from the modified triple-axis spectrometer are in good agreement with those data from the 30 m NG3-SANS instrument located at the National Institute of Standards and Technology (Gaithersburg, MD, USA).

## 1. Introduction

Small angle neutron scattering (SANS) has proven a powerful technique for the study of molecular structures and morphologies with length scales ranging from 10 to 1000 Å. Dedicated SANS instruments normally cover a scattering vector range ($q$ range) from 0.001 to 0.6 $Å^{-1}$, where $q$ is defined as $(4\pi/\lambda)\cdot\sin(\theta/2)$, where $\lambda$ and $\theta$ are the neutron wavelength and the scattering angle, respectively. Measuring the lowest possible $q$ value, $q_{\min}$, usually requires long wavelength neutrons and a small incident beam. Long wavelength neutrons are usually produced from a cold neutron source, shifting the thermal neutron energy distribution toward lower energies. A small incident beam is generally produced using either a highly collimated or focused [2] neutron beam. Using a velocity selector, which increases the incident neutron bandwidth ($\Delta\lambda/\lambda$), can greatly compensate the reduced incident neutron flux caused by the restricted collimation.

Presently, the Canadian Neutron Beam Centre (CNBC) does not have a cold source to produce long wavelength neutrons. Therefore, we resorted to adapting a triple-axis spectrometer capable of carrying out SANS measurements. However, in order to so a number of technical obstacles had to be overcome. Incident neutron wavelength is selected through the use of a single crystal monochromator ($\Delta\lambda/\lambda \sim 1\%$), instead of a velocity selector ($\Delta\lambda/\lambda \geq 10\%$). One of the methods used to increase incident neutron flux, while not increasing the beam size, is to employ multiple incident beams that converge at a single spot on the detector. This concept of one-dimensional confocal beams was first proposed and tested by Nunes [3] and was later developed for two-dimensions (2D) by Glinka, *et al.*[4]. At the CNBC we have designed and implemented a confocal Soller collimator (CSC) for use at the N5 triple-axis spectrometer, making it suitable for SANS measurements, where $q_{\min}$ of 0.006 Å$^{-1}$ is achievable. This development allows for the ubiquitous triple-axis spectrometer to double, with minimal cost and effort, as a capable SANS instrument. Due to the slit geometry on the scattered side, additional horizontal Soller collimators (HSCs) are sometimes required to reduce the smearing due to vertical divergence. The comparison between the N5 data and those collected at the 30 m NG3-SANS instrument located at the National Institute of Standards and Technology (NIST) shows excellent agreement.

## 2. Instrument Configuration

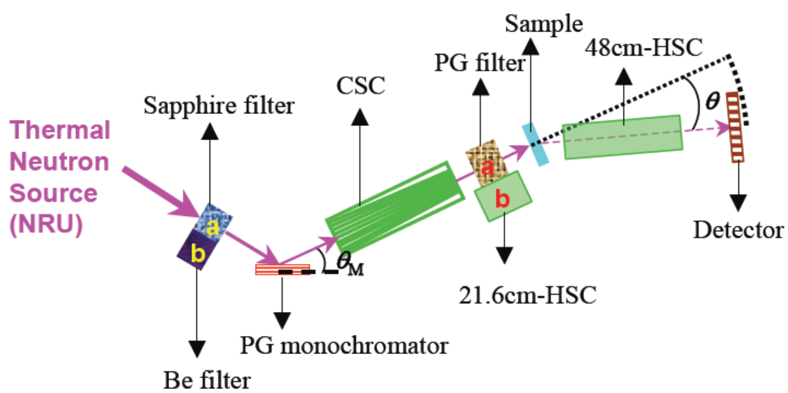


**Figure 1.** Schematic of the N5-SANS. The widths of the CSC are 0.13 cm and 0.1 cm at the monochromator and sample ends, respectively. The height of individual channels of both HSCs is 0.25cm.

Figure 1 shows a schematic of the N5-SANS configuration. λ was selected using the (002) reflection of a pyrolytic graphite (PG) monochromator, from which three λs (i.e., 2.37, 4, and 5.23 Å) were selected in the current setup. The higher order harmonics of the fundamental neutron wavelength (i.e., λ/2, λ/3, etc.) are reduced through the use of either a beryllium (Be) (for λ > 3.99 Å) or a PG filter (for λ =2.37 Å) cooled by liquid nitrogen. A sapphire filter is optionally used for reducing the presence of "fast neutrons" [5]. Depending on the desired wavelength, either a sapphire (for λ < 3.99 Å), or Be (for λ > 3.99 Å) filter is placed upstream of the PG monochromator (Fig. 1). Selected neutrons then go through the CSC (length $L_{\mathrm{CSC}}$ of 66 cm) which is made up of 23 channels, whereby each channel is separated by steel blades coated with $Gd_2O_3$. All channels converge at the same spot on the detector. After the CSC, neutrons go through either a PG filter, or in the case of 4 and 5.23 Å neutrons, a 21.6 cm long HSC. The neutrons then interact with the sample, and the scattered neutrons then go through another 48 cm long HSC. A 32-wire $^3$He position-sensitive detector is placed after the second HSC with an effective sample-to-detector distance, $D_{\mathrm{SD}}$, of 1.43 m. Each wire is capable of detecting scattered neutrons at the corresponding θ.

## 3. Data Reduction

The reduced scattered intensity $I_{RED}$ $(q)$ is obtained through a standard data reduction procedure applied to the raw scattered intensity, $I_{\mathrm{RAW}}$ $(q)$ through the following equation.

$$I_{RED}(q) = \frac{\left[I_{RAW}(q) - I_{BGD}(q)\right]}{T_{SAM}} - \frac{\left[I_{EMP}(q) - I_{BGD}(q)\right]}{T_{EMP}}, \tag{1}$$

where $I_{BGD}(q)$, $I_{EMP}(q)$ are the background (blocked beam) and empty cell scattering, respectively, and $T_{SAM}$ and $T_{EMP}$ are the sample and empty cell transmitted intensities, respectively.

## 4. Results

### 4.1. Characterization the CSC

Figure 2 shows a comparison of the total intensity (all channels open) with that from individual channels. When fitted with a Gaussian function, the centers of the peak positions vary within 0.03° from each other, an indicator of each channel's alignment. The total incident flux from the 23 channels is approximately 20 times that from an individual channel (ideally 23 times).

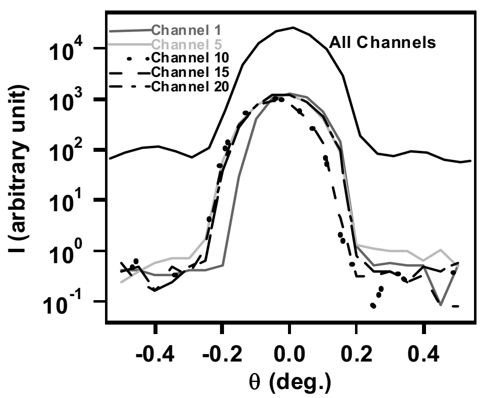


**Figure 2.** Comparison of the incident beam profile for all channels open compared to individual channels.

### 4.2. The resolution function

A SANS instrument's resolution function depends on its geometry (e.g., pinhole, slit, etc.). In slit geometry, scattering from isotropic samples is smeared due to vertical divergence. Therefore, the use of HSCs is sometimes needed.

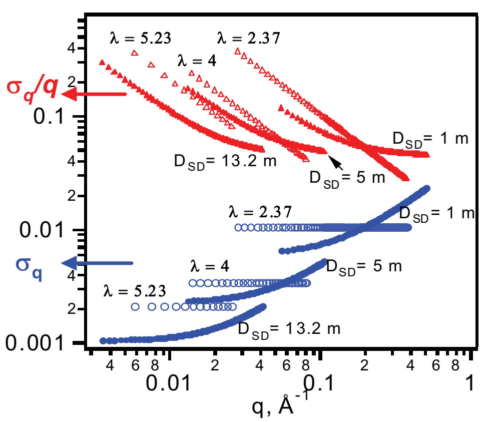


**Figure 3.** The instrumental resolution of the N5-SANS and NG3-SANS instruments. Both $\sqrt{\sigma_q^2}/q$ (red triangles) and $\sqrt{\sigma_q^2}$ (blue circles) are plotted as a function of $q$. Hollow and solid symbols represent N5-SANS and NG3-SANS data, respectively.

The resolution function $\sigma_q$ for the slit scattering geometry was derived by Mildner and Carpenter [6,7]. Figure 3 shows a comparison of the calculated $\sigma_q$ for the N5-SANS and a pinhole geometry instrument (i.e., the NG3-SANS located at the NIST Center for Neutron Research) [8]. NG3-SANS' $\sigma_q$ is calculated based on a neutron wavelength of 6 Å, a $\Delta\lambda/\lambda$ of 15% and three $D_{SD}$s (1, 5 and 13.2 m). For NG3-SANS, $\sigma_q$ (Fig. 4) increases at higher $q$ values as a result of a poor $\Delta\lambda/\lambda$, eventually surpassing the gains from distance collimation. However, this increase in $\sigma_q$ does not affect the smearing effect as $\sigma_q /q$ continues to decrease with increasing $q$. In the case of the N5-SANS, $\sigma_q$ plateaus for a given λ because the main contribution is from distance collimation. At low $q$ ($q$ <0.05 Å$^{-1}$) the instrumental resolution, $\sigma_q /q$ of the N5-SANS is worse than the NG3-SANS, but comparable and sometimes even better at $q$ > 0.1 Å$^{-1}$.

### 4.3. Experimental Results

Two standard polystyrene microsphere (diameters of 24 nm and 50 nm from Bangs Laboratories) solutions of concentration 1 wt.% in $D_2O$ were examined. The absolute intensities for both samples are using the N5-SANS and NG3-SANS are shown in Fig. 4(a) and (b). The N5-SANS data are rescaled for each $q$-range and then re-plotted on the same figure. The $q_{min}$ of N5-SANS data is found to be ~0.006 Å$^{-1}$. Slight discontinuities in the data are observed in the overlap regions, presumably due to different resolution functions associated with the different $q$-ranges. We find excellent agreement between the NG3-SANS and N5-SANS data proving the successful implementation of this SANS design.

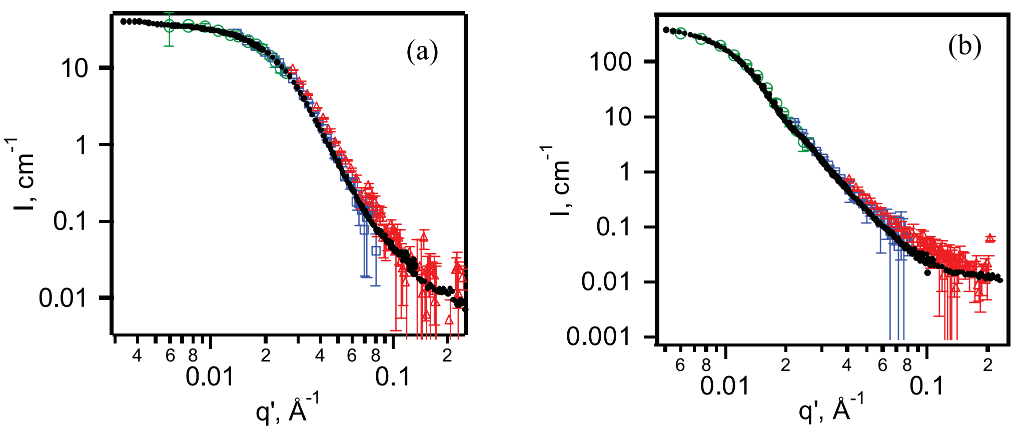


**Figure 4.** SANS data from 1 wt.% microspheres of diameter (a) 24 nm and (b) 50 nm obtained from the N5-SANS (open symbols) using $\lambda$ = 5.23 (circles), 4 (squares), and 2.37 (triangles) Å and the NG3-SANS (solid circles).

## 5. Future Work

As a result of this experience, CNBC is now constructing a dedicated SANS instrument using a 2D image plate detector. The success of the N5-SANS has validated the concept of the CSC, which will now be applied to the design of a composite 2D-CSC, effectively two orthogonal 1D-CSCs. As a result, the HSCs will not be required.

## References

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